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ABRASION-RESISTANT ALUMINIZED-COATED ARAMID FABRICS FOR MANUFACTURE OF FIREFIGHTERS' PROTECTIVE CLOTHING

NAVY CLOTHING AND TEXTILE RESEARCH FACILITY
NATICK, MASSACHUSETTS

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TECHNICAL REPORT NO. 160

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 160	2. GOVT ACCESSION NO. AD-A157056	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) ABRASION-RESISTANT ALUMINIZED-COATED ARAMID FABRICS FOR MANUFACTURE OF FIREFIGHTERS' PROTECTIVE CLOTHING		5. TYPE OF REPORT & PERIOD COVERED Final Report; 31 July 84 6. PERFORMING ORG. REPORT NUMBER 88860
7. AUTHOR(s) Richard Brenneman		8. CONTRACT OR GRANT NUMBER(s) N00140-83-C-RA12
9. PERFORMING ORGANIZATION NAME AND ADDRESS ARTHUR D. LITTLE, INC. Acorn Park Cambridge, MA 02140		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 62758N, 58523, ~~523-091~~, 62100
11. CONTROLLING OFFICE NAME AND ADDRESS Navy Clothing & Textile Research Facility 21 Strathmore Road Natick, MA 01760-2490		12. REPORT DATE May 1985 13. NUMBER OF PAGES 68
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) Same as 11.		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

Same as 16.

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Abrasion-Resistant Aluminized-Coated Aramid Fabrics; ~~Firefighters' Protective Clothing;~~ Electron-Beam Cured Coating; Aluminized Mylar; Kapton Film; ~~Kevlar Fabric;~~ Military Protective Clothing.

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Under contract with the Navy Clothing & Textile Research Facility, Arthur D. Little, Inc. has developed and evaluated two experimental abrasion-resistant aluminized-coated aramid fabrics for use in firefighters' protective clothing. The first fabric contains an electron-beam cured coating applied to both sides of the aluminized mylar used in the existing laminated fabric. The second one contains Kapton film, which is aluminized on one side and laminated on the metal side to a Kevlar fabric.

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ABSTRACT (continued)

Although each experimental fabric has better abrasion resistance than the existing aluminized fabric, each represents a compromise between longevity of performance and efficiency of reflectivity over a wide spectrum of infrared energy. (U)

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SUMMARY

The required yardage of two experimental approaches to developing an abrasion resistant fabric for firefighters protective clothing has been delivered to NCTRF, Natick, MA, for fabrication into uniforms.

The first is an electron-beam (EB) cured coating on the two-side aluminized mylar used in the existing laminated fabric. The second is a one-side aluminized Kapton (K) film laminated metal side to Kevlar fabric.

Both experimental products greatly outperform the existing aluminized fabric in abrasion resistance. The color photograph included in the Summary illustrates relative abrasion resistance in comparison to the existing product.

However, neither of the experimental products will pass all of the specified tests.

The EB product, in spite of its superior abrasion resistance, will not pass the specified tape peel test for coating adhesion. In view of the adhesion of the EB coating during a more severe abrasion test than specified, we question the significance of the specified tape peel test for coating adhesion. In addition, a commercial supplier probably could improve tape peel resistance of the EB coating by proprietary technology.

The Kapton product will not pass the specified wet flex test because the film parts from the reflective aluminum leaving the metal on the delaminated fabric. The wet delamination is believed to occur because the Kapton film absorbs about two percent moisture during the immersion period.

The Kapton product illustrates the principle that a high temperature melting point film, aluminized on one side and laminated metal side down to Kevlar fabric, can provide abrasion and chemical resistance as well as reflectivity. However, its water absorption and adhesive characteristics severely limit use in the water-prone firefighters environment.

We believe other high-temperature melting resins, such as polysulfone or polyether-imide, would probably have more suitable adhesive properties than Kapton because they are more water resistant.

Cont -

Unfortunately, such alternative materials were not available as suitable thickness films for the application.

Both the experimental EB and Kapton products absorb more heat than the existing product because they reflect less infra-red energy than the pure aluminum on the existing product. It is a fundamental fact that only gold metal can be coated on aluminum without reducing its reflectivity of the aluminum. All other materials, and particularly organic polymers, will reduce reflectivity when placed over aluminum.

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CONCLUSIONS

1. The two products, EB and Kapton, represent a compromise between longevity of performance and efficiency of reflectivity over a wide spectrum of infra-red energy.

2. The EB product can be commercialized by any interested supplier using state-of-the-art technology.

3. The Kapton product should not be commercialized because its inherent water absorption tends to self-defeat any adhesive system.

4. We believe there are deficiencies in the present test methods.

5. We believe there are short-comings in the existing full uniform construction.

RECOMMENDATIONS

1. Test Methods

   a. The present abrasion test method should be replaced with the "Accelerated Wyzenbeck Test" described in the report.

   b. The present subjective IR evaluation method should be replaced with the quantitative calorimeter test described in this report.

2. High-Temperature Resistant Films for Reflective Laminates

   Further research should be directed toward high-temperature resistant materials that are emerging commercially as thin films. Such films have the potential to overcome the adhesion deficiency identified in the Kapton experimental product. Because high-temperature

resistant polysulfone, polyethersulfone and polyether-imide polymers have low water absorption they probably will not be subject to the metal delamination incurred by Kapton in a wet environment.

We believe that the market for Kapton-like materials will stimulate the commercial research necessary to extrude films thin enough for fabric lamination. Indeed, a Naval application research program for firefighters uniforms may accelerate progress in thin films if commercial vendors are aware of such a need.

3. Construction Alternatives for Firefighters Uniforms

A research program should be initiated to review the neoprene-coated "rain coat" layer of the existing uniform. That garment, particularly the nylon fabric, represent a thermal weakness in the total construction. The nylon fabric melts and welds itself to the underlying quilted fabric when the outermost laminate fails by abrasion. The black neoprene coating then absorbs sufficient radiant energy through the damaged reflective laminate to melt the supporting nylon fabric.

CHARRED LAMINATE
MELTED RAINCOAT

NO CHAR OR VISIBLE
DAMAGE TO RAINCOAT

CONTROL EB COATED KAPTON

ABRASION RESISTANCE

68860

ABRASION RESISTANCE ($3000W_A$)
VS
INFRA-RED TEST

FIG. 1 S-1

I. EXISTING PRODUCT

An understanding of the existing product is essential to comprehending the research efforts and the concepts that were developed in the course of this assignment.

A. CONSTRUCTION

An exploded view of the cross section of the existing metallized fabric is shown in Figure I-1. Each of the subsections that comprise the finished aluminized fabric represents commercially available materials that require different technologies and skills. The three subsections are combined or laminated by a fourth party, such as Gentrex Corporation, to produce a product that meets the specifications of MIL-C-87076 (hereafter referred to as the specification).

The skills of the laminator and the adhesive are proprietary to each laminate manufacturer. Although the source and identity of Subsections A and C are reasonably well known, the manufacture of the double metallized film and the Kevlar fabric undoubtedly also involves proprietary skills and knowledge. Therefore, our research involved considerable empirical work with commercially available products rather than precise scientific experimentation with materials of known composition.

Our efforts focussed mainly on Subsection A, the reflective layer(s), although we identified an adhesive that allowed us to simulate the existing product and test it for the primary functions--infrared, abrasion and chemical resistance. The Kevlar fabric was used as received throughout the work.

A second aluminized fabric, 3M's transfer fabric, was briefly considered in the early stages of the work. After discussions with the technical contract monitor, however, it was agreed to suspend further efforts with the product until suitable new materials and skills were identified for the fabric described in Figure I-1. All subsequent discussion will refer to the laminated fabric of Figure 1.

B. PERFORMANCE

The laminated fabric, in its virgin condition, provides excellent protection against radiant heat for a limited period. However, because the reflective layer absorbs some radiant energy there is a heat buildup in the fabric as a function of exposure time. We have been informed that the virgin material affords sufficient protection for about 30 seconds of exposure to a high-radiation-intensity fire.

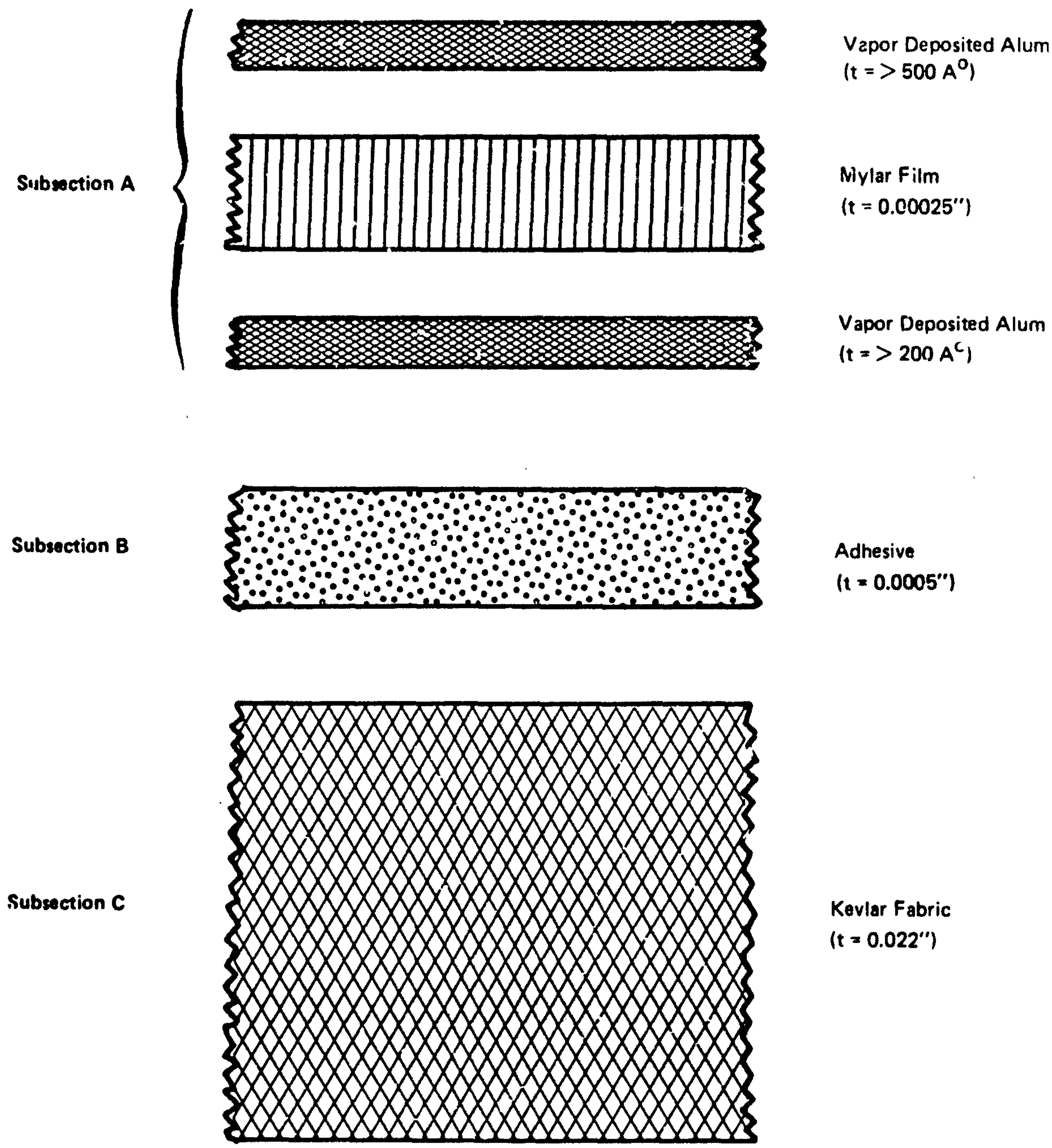


FIGURE I-1 CONSTRUCTION OF EXISTING FABRIC

However, field performance shows the reflectivity of the fabric is degraded by abrasion and/or chemical attack of the top layer of vapor deposited aluminum. As a result of mechanical and/or chemical wear, the article becomes unsightly and less protective. The end result is a potentially unacceptable replacement rate of the firefighters uniform.

C. REFLECTIVITY OF MATERIALS

A hot fire, defined by the specification as one that reaches a temperature of 2730°F, emits a wide band of electromagnetic energy which peaks at about 1.2 microns but extends from the visible, 0.4-0.8 micron, to above 7.0 microns. About 95% of the above radiant energy occurs within the 0.4 to 7.0 micron range.

Metals, such as aluminum or gold, will reflect over 90% of the specified radiant energy and heat up comparatively little over the specified exposure time of 30 seconds. That is why the virgin metallized fabric is effective.

However, organic materials, such as the Mylar film used to support the thin layer of reflective aluminum, will absorb much of the specified radiant energy, particularly at selected wavelengths, and get hot enough to melt or decompose given a sufficient exposure. Therefore, even a small loss of surface reflective material will degrade the performance of the firefighter's fabric.

Conversely, placing a layer of any material, except more reflective gold, over the aluminum to protect it against mechanical or chemical loss, will reduce overall reflectivity and allow more heat to be absorbed in the laminate.

Therefore, compromises between longevity of the product and rate of heat buildup are inherent. In other words, any protective layer for the aluminum will cause heat to build up more rapidly, but because the thin reflective aluminum is extremely susceptible to mechanical and chemical loss, such protective layers offer an opportunity to delay a potential catastrophe to the firefighter.

II. MECHANISMS OF WEAR OF THE EXISTING PRODUCT

A. CHEMICAL

Any oxidizing chemical will completely destroy the reflective characteristic of the top layer of aluminum within some reaction conditions. For example, a 4% sodium hydroxide solution will completely destroy the top layer of aluminum in seconds. Other chemicals, such as the potasium carbonate used as a dry fire extinguisher, will also destroy reflectivity (over a longer period), particularly in a humid environment. Indeed, even the acidity from a finger print will gradually remove the external layer of aluminum.

Although external chemical wear seldom affects the underlying Mylar, the second layer of underlying aluminum, the adhesive or the fabric, the laminate has greatly reduced infrared resistance in the area of chemical wear of the outer layer of aluminum. Inspection of chemically worn areas by transmitted light clearly shows loss of surface aluminum. Infrared testing of such chemically worn areas, as described in MIL-C-87076, shows that enough radiant energy penetrates the fabric to scorch the backup blotter and even the Kevlar fabric in less than 30 seconds. While the fabric has the heat capacity to absorb the energy transmitted by very small areas (i.e., less than 1/8" diameter) and prevent blotter scorch behind the chemically distressed area, there is, nevertheless, a small hot spot. And if the chemically distressed area is large, any combustible material behind the fireproof Kevlar fabric is likely to char or burn.

Observations of transmitted visible light behind a chemically distressed area, during the infrared test, indicate that a change of state occurs during the test. The transmitted light gradually grows brighter for several seconds and then appears to rapidly increase in size and intensity. Indeed, the size of the chemically distressed area will expand during the infrared test. For example, a 1.5-cm circular spot abraded with a pencil eraser will cause visible damage to a 3.5-cm circular spot during the IR test.

We believe that the Mylar film melts and disrupts the second reflective layer of aluminum, thus allowing radiant energy to heat up or scorch any material behind the aluminized fabric. Even if the blotter in the specified test does not contact the laminate, it will scorch because of radiant energy passing through the distressed area in the aluminum.

Subsequent work with higher-melting-point films which have been metallized on one side, shows that Kapton, Ultem, and Victrex films will protect the underlying reflective aluminum layer because they do not melt.

B. THERMAL FAILURE

Heating the aluminized fabric close to or above the melting point of mylar, about 250°C, will destroy the reflectivity of both aluminized layers in the absence of any mechanical force. Therefore, any contact with a hot conductive surface (about 250°C) will destroy the utility of the garment.

C. MECHANICAL

1. Abrasion

The abrasion specification defines a cotton duck cloth, a weight and a reciprocating cycle to abrade the specimen.

Within those conditions, the texture of the aluminized fabric is a primary determinant of total wear because only the high points of the underlying twill fabric contact the abradant. The reflective aluminum is rapidly worn off the Mylar at the contact areas but remains untouched in recessed areas as shown by inspection, using transmitted light at 300 and 900 abrasion cycles. After the aluminum is worn off, the high points of the exposed Mylar become the abrasion resistant material.

Increasing the loading beyond that specified greatly accelerates the apparent wear rate of the laminate because the high points of the aluminized fabric are flattened somewhat. However, pressures beyond that in the specified Wyzenbeck test are required to achieve uniform contact with all of the aluminum surface. Similar results are obtained using the Taber or the Stoll tests--neither one provides the pressure necessary for full contact with the contoured aluminum surface.

A brush, even a "medium" nylon bristle toothbrush, will wear the aluminized fabric surface faster than the specified test with cotton duck cloth. Such a toothbrush, mounted on the Wyzenbeck tester with no load on the lever arms, will remove almost all the external aluminum from the aluminized fabric just as fast as the specified test removes aluminum only from raised areas of the fabric. A "falling sand" test will remove all the metal from the aluminized fabric even more quickly than the specification test does only at the peaks of the fabric.

2. Creasing, Stretching and Flexing

These forces cause localized loss of reflectivity as judged by transmitted light. We believe the effect to be caused by the difference in modulus between Mylar and metal. That is, the metal will not survive

those deformations easily tolerated by the plastic. Such deformations of the aluminized fabric are inevitable in clothing, particularly at elbow and knee areas.

3. Microscopic Examination of Wear Patterns

An examination of wear patterns at up to 400 magnifications suggests the following sequence of events:

- While random surface scratches occur almost instantaneously in any abrasion test, mud cracking of the aluminum on the high points of the fabric is almost inevitably the first sign of failure;

- The mud cracked aluminum flakes off or is sheared off the plastic substrate in a geometrical pattern that corresponds to the original mud crack pattern;

- Mud cracking occurs readily when the fabric is briefly flexed or stretched by hand; and

- Fabrics that have been flexed or stretched are abraded faster than virgin fabric.

4. Projected Mechanism of Failure

It is apparent that the current product fails or loses its desired level of reflectivity by a variety of individual or combinations of chemical, thermal or mechanical effects. Fundamentally, however, the physical properties of the metal and the carrier plastic are almost antagonistic when the composite is stressed within its elastic limit. Aluminum cannot be expected to survive as a film within the physical stresses and strains easily accommodated by the tough laminate--that is, the metal will rupture and mud crack long before the plastic or laminate even becomes fatigued.

After the metal has mud cracked that very small local area is held to the plastic only by electrostatic van der Waals or London forces because the two are not chemical bonded. In fact, the literature suggests that a layer of metal oxide may exist between the metal and the plastic.

Finally, it is clear that even minimal mechanical stress is sufficient to remove the small areas of metal defined by the mud cracks that are induced by small deformations of the laminate.

III. ALTERNATIVE METALS

A. EXPERIMENTAL WORK

1. Materials

We investigated other metals and combinations of metals either as the sole metal reflective layer or as additional coatings on top of aluminum. Mylar film was used as the carrier in these experiments. These data are found in Table III-1.

In these experiments, the aluminized films were laminated to Kevlar fabric using a high-temperature-resistant, non-flammable, solid-film adhesive. The adhesive was adequate for laboratory testing of radiation and abrasion resistance, but not suitable for commercial use because its peel strength is inadequate.

The laminates were first tested in virgin condition for resistance to the specified infrared test. If they survived that test they were tested for abrasion resistance using the accelerated Wyzenbeck test described in Section VII. For controls, we used both the commercial Gentex material and a laboratory laminate of double aluminized Mylar (supplied by Metallized Product Company) to Kevlar fabric.

The peak temperatures behind the fabrics were recorded with a thermocouple. This technique was used to support the visual scorch observation required by the specifications. The test data are shown in Table III-1.

Clearly, increased metal hardness and thickness do not provide an answer to abrasion resistance.

2. Other Metallization Processes

Because vapor deposited metal hardness and thickness do not significantly affect abrasion resistance, other metallizing processes were considered. Little experimental work was carried out with products metallized by different methods because many of the processes are not suited to metallizing the quantity of film required for our deliverable products.

a. Chemical Metallization

Gold, silver, nickel, etc. can be chemically deposited on plastic film as an opaque reflective layer. We tested the effect of chemically deposited gold on plastic film.

TABLE III-1

ALTERNATIVE METALS

Metal	Infrared Resistance (Temperature, °F of Behind Fabric)		Remarks
	Virgin	Abraded, 300 Cycles	
Aluminum	200	Scorch	Gentex control
Aluminum	235	Scorch	Laboratory control
Aluminum	290	Scorch	Heavy aluminum deposit (3000A)
Germanium/Aluminum	320	Scorch	Hard germanium on top of aluminum
Germanium	Scorch	-	Much harder than aluminum
Chromium/Aluminum	Scorch	-	Hard chromium on top of aluminum
Chromium	Scorch	-	Much harder than aluminum
Nickel	Scorch	-	Much harder than aluminum
Cobalt/Nickel	Scorch	-	Much harder than aluminum
Nickel/Iron	Scorch	-	Much harder than aluminum
Iron	Scorch	-	Much harder than aluminum
Gold	200	Scorch	Approximate hardness of aluminum
Silver	200	Scorch	Harder than aluminum

Chemical deposition does not improve abrasion resistance. Chemically deposited gold wears as fast or as badly as vacuum deposited gold. Similar results were obtained with the other chemically deposited metals.

b. Other Physical Methods of Metallization

Over the past several years new trends have developed in vacuum metallizing. Some are refinements of the older resistance or induction heating process. Others have grown out of the semiconductor industry and are being geared to metallize flexible plastic webs. Some of these processes can also apply organic materials to a metallized film; these are discussed in a subsequent section.

Although these new processes imply better quality of metallization for electrical purpose or for information storage applications and/or better economics, none promise a more durable metal film or better reflectivity.

(1) Electron-Beam Evaporation. Electron-beam evaporation is a refinement of the standard vacuum evaporation process in that the coating material is heated to evaporation and subsequently condensed on the substrate surface. Rather than using resistance or induction heating, the coating material is scanned by a high energy, focused, electron-beams, and only the surface is evaporated. The controlled energy generated by the electron beam allows the higher melting materials (metals, metal oxides and refractories) to be deposited with excellent process control and reduced potential for substrate damage.

(2) Sputter Coating. This method generically refers to those deposition processes wherein a thin film is formed on a substrate by the condensation of neutral atoms which have been removed from a target material by impinging ions. Typically, the vacuum chamber is evacuated then backfilled with a gas (usually argon) in which a glow discharge can be initiated and maintained. The target, a metal plate coated with the material to be deposited or from which a deposit will be fabricated, is connected to a negative voltage source (dc or rf) and therefore is the cathode in the glow discharge "circuit". The position of the anode is variable, depending upon the desired result, but is usually lateral to the cathode. The substrate is made to pass close to the cathode, and may be supported by a cooling roll.

As the glow discharge plasma is initiated, positive ions of the resident gas strike the cathode and remove neutral atoms of the cathode surface by momentum transfer. These neutral atoms then condense into a thin film on the substrate surface. Other charged particles and radiation are also produced at the cathode surface and may profoundly influence the quality of the substrate coating. These include secondary electrons and ions, desorbed gases, x-rays and photons. If these emissions are allowed to strike the substrate, considerable heating or etching may occur, which, in some processes, may be desirable. Excessive outgassing by the substrate may contaminate the plasma and seriously affect the sputtering rate and the quality of the deposited coating.

Frequently, a mixture of gases (e.g., argon and oxygen) is used to form a compound of the target material and the reactive gas. This process is called reactive sputtering, and is commonly used to form coatings of indium-tin oxide and titanium dioxide for window films.

(3) Magnetron Sputtering. Another refinement of the sputtering process is the use of a magnetic field to confine the plasma. This process, called magnetron sputtering, increases sputtering efficiencies and therefore deposition rates.

B. CONCLUSIONS

The abrasion resistance of vapor deposited aluminum is not a function of metal thickness.

Harder metals are of no interest in the application primarily because they absorb enough heat to destroy the composite. Furthermore, harder metals do not increase the abrasion resistance when deposited over aluminum. Finally, neither gold nor silver, although effective reflectors, offer advantages over less expensive aluminum.

IV. COATINGS FOR THE EXISTING ALUMINIZED MYLAR

The metallized plastic industry has been concerned with protecting aluminized plastics since the beginning of the industry. Many of the suppliers have installed coating facilities for this purpose. Indeed they have met or exceeded many industrial requirements for abrasion at ambient conditions. While those commercial coatings are more or less effective for maintaining appearance in an ambient environment, no substantial thought has been given to thermal resistance as defined by the firefighters application. Indeed, there are few clues in the literature specific to this problem.

The specification for reflective firefighters fabric implies heat resistance when exposed to an infrared source radiating 1.9 calories per second per square centimeter. Because any coating material that absorbs the radiant energy will get hot, stability of the coating material under these conditions becomes paramount. Just how hot the coating becomes is a function of its thickness and its specific infrared absorption characteristics.

One measure of "how hot" exists in the commercial product because the Mylar film (M.P. 250°C) is the weakest thermal component of the laminate -- aluminum and Kevlar fabric melt or decompose at about 1200°F and 600°F, respectively.

When the top reflective layer of aluminum is removed from the commercial laminate it fails the specified infrared test. Under those conditions we measure a temperature of >500°F in the adhesive layer behind the double aluminized film. When the free-standing double-aluminized mylar film is exposed to convection or conduction heat at a temperature of 450-480°F, the Mylar melts and the laminate loses all its shiny reflective metallic appearance.

A. CONVENTIONAL COATINGS

The metallized-film industry has been confronted with the fragility of vapor deposited aluminum coating since it started to market products. A wide range of thermoplastic acrylic, vinyl chloride, cellulosics, etc. has been successfully applied to aluminized film for mechanical protection at ambient conditions without regard to thermal conditions that normally would degrade the coating or the supporting film. Some of the thermoplastic materials have also been hot melt or extrusion coated. For example, many packaging pouches or bags consist of metallized Mylar extrusion coated with polyethylene, propylene, nylon etc. Furthermore, some metallized Mylar has been coated with conventional thermosetting polymers where curing conditions are not harmful to the supporting film.

We tested a number of commercially coated metallized Mylar films (laminated to Kevlar) that were reported to have abrasion resistance. All of these coated samples melted rapidly or charred in the infrared test and allowed the radiant energy to scorch the underlying Kevlar and the backup blotter.

Further confirmation that high-temperature-resistant coatings are needed was obtained by coating double-aluminized Mylar film with various conventional coatings that are known to be abrasion resistant at ambient conditions.

The data in Table IV-1 show that these materials do not have the required thermal resistance. All the virgin or unabraded coatings charred, burned or decomposed when exposed to the specified IR energy.

Evidently, the radiant energy of the specified test is sufficient to heat these materials to the point where the chemical bonds between atoms are destroyed. In addition to causing burning, i.e., oxidation of the surface coating, sufficient heat and/or reaction with the top aluminized layer on the Gentex film caused the supporting Mylar film to melt. As a result, radiant energy penetrated the Kevlar fabrics and charred or burned the backup blotter.

Ordinary polymers cannot maintain their rigidity, shape, strength or chemical integrity at temperatures much above 400°F. At these temperatures ordinary polymers are fluids and/or rapidly decompose in the presence of oxygen. The relatively weak covalent bond that connects one monomer segment to another in ordinary polymers cannot withstand the thermal and oxidative stresses at 400°F.

B. NASA COATED ALUMINIZED KAPTON

We tested samples of this material, which were supplied by National Metallized Co., for abrasion and IR resistance. The coatings and the underlying aluminum layer wore off the Kapton film in the specified Wyzenbeck test. After the abrasion test they failed badly in the IR test; that is, the test blotter ignited and burned.

C. HIGH-TEMPERATURE SOLUTION COATINGS

Because of new technology requirements for light, strong and formable materials that can withstand temperatures above 400°-500°F, newer solution-coatable organic polymers have evolved. Such materials are described as polysulfones, polyether sulfones, imides, imide-amides, ether imides, etc.

We investigated several types and classes of high-temperature solution polymers by coating the Gentex fabric at several coating weights.

TABLE IV-1

CONVENTIONAL POLYMER COATINGS

	Material	Chemical Type	Performance in Infrared Test - Not Abraded
solution coating	Acryloid A21	Acrylic	Burns
	Acryloid B72	Acrylic	Burns
	Mobay	Urethane	Burns
	VYHH	Vinyl Chloride	Burns
	Cymel 1158	Melamine	Burns
	SR 182	Silicone	Decomposed to white powder
	SR 120	Silicone	Decomposed to white powder
	Extrusion Coating	Nylon	Burns
	Extrusion Coating	Polyethylene	Burns
	Extrusion Coating	Polypropylene	Burns

1. Thermoplastics

Several types of high-temperature-resistant polymers are coated on aluminum cookingware to provide scratch and abrasion resistance and to prevent fried foods from sticking. Such polymers do not provide a continuous film upon evaporation of solvent. They must be baked, fused and annealed at temperatures above 500°F for more than several minutes to obtain a continuous film that adheres to aluminum. Clearly, these temperatures exceed the thermal capability of Mylar. We find that the reflectivity of Gentex fabric is largely destroyed under the baking schedule recommended by the manufacturer of such coatings.

However, we were able to fuse some coatings on Gentex fabric with infrared radiation because its reflective properties protect the Mylar film from melting. Conversations with the coatings manufacturer revealed that such radiation curing of such materials is an uncertain art and generally does not provide adequate adhesion to aluminum metal--both time and temperature are necessary for adhesion.

The data in Table IV-2 indicate that a number of these materials may have adequate thermal resistance. However, none of the coatings increased the abrasion resistance of the laminate.

The abrasion resistance problem was found to be due to lack of adhesion of the coating to the aluminum metal. In all cases the coating could be easily removed with Scotch tape. Microscopic examination showed that the coatings were stripped off the aluminized fabric in the initial stages of the Wyzenbeck abrasion test.

Because some of these materials appear to have adequate thermal properties, we investigated various ways of cleaning or etching the aluminum surface of the laminate or priming the surface with ilicone adhesion promoters.

While some of the pretreatment and curing cycles looked promising, as judged by the Scotch tape test, all such coatings continued to strip off the aluminum under the shearing forces of the Wyzenbeck abrasion test.

2. Thermoset Coatings

All of the thermoset polyimides require curing for at least 30 minutes at 500°F or even higher temperatures after the solvents are removed by evaporation. Because the Gentex fabric will not withstand these conditions, we performed very little experimental work with thermoset polyimides. Incomplete cures at 300°F produced only a brittle or mud-cracked coating which could be wiped off the aluminum surface with lens tissue.

TABLE IV-2

HIGH-TEMPERATURE SOLUTION COATING

Coating Chemical Identity	Infrared Resistance: Virgin Condition	Infrared Resistance: Modified Wyzenbeck Cycles to Failure
None - Control	OK	3-600
Polyether imide	OK	3-600
Polyimide	OK	3-600
Polyether sulfone	Fails	
Silicone/alkyd	OK	3-600
Polytetrafluoroethylene	OK	3-600
Polysulfone	OK	3-600
Silicone	OK	3-600

D. ELECTRON-BEAM (EB) CURABLE COATINGS

These coatings are formed by curing or crosslinking a complex mixture of multifunctional acrylic monomers and oligomers with high- energy electrons. The highly crosslinked polymers are reported to be transparent, tough, abrasion resistant and flexible in thin films. While EB coatings have no history of thermal resistance (as defined by the specification) Metallized Products is applying them to various metallized Mylar films for abrasion resistance at ambient conditions.

1. Coating Formulations

The proprietary EB coatings are formulated by a limited number of companies who supply the equally limited number of EB coating facilities. Mobil Chemical Company will not divulge compositions other than identify them as radiation curable acrylic compounds for abrasion resistance on polyester film. Similar responses were encountered with other EB coating suppliers.

In essence, an EB coating is a solution of a multifunctional vinyl prepolymer or oligomer in multifunctional acrylic monomers. In general, the industry has found acrylics to be the most widely useful vinyl systems because of their fast polymerization rates. The composition may be modified with monofunctional monomers, such as N-vinyl pyrrolidone, to control viscosity. Within these parameters many combinations of materials are feasible and the exact compositions are tailored for the coating method and the final product requirements.

The liquid coatings are polymerized by being exposed to electrons at 100,000 to 300,000 volts.

2. Electron-Beam Curing Equipment

Energy Sciences, Woburn, MA, manufactures equipment for EB curing of flexible webs, i.e., their Electrocurtain®.

The source of electrons in the Electrocurtain-type EB processor used in most converting applications (see Figure IV-1) is a heated filament (or cathode) inside the electron gun. The beam of electrons (or current) is formed by the gun cylinder after the filament temperature is raised to some fixed level inside the vacuum chamber. A beam or curtain of electrons from it is then accelerated by the electric field between the electron gun-structure terminal and the outer wall of the chamber. This voltage, which can range from 100,000 to 300,000 volts depending on the system, determines the penetration capability of the electrons which emerge through the metallic foil window and into the product. The "transparent" foil window is capable of supporting one atmosphere of pressure while at the same time intercepting only a small percentage of the beam power. The whole operation is performed within the integral shielding system or "Selfshield" to confine and

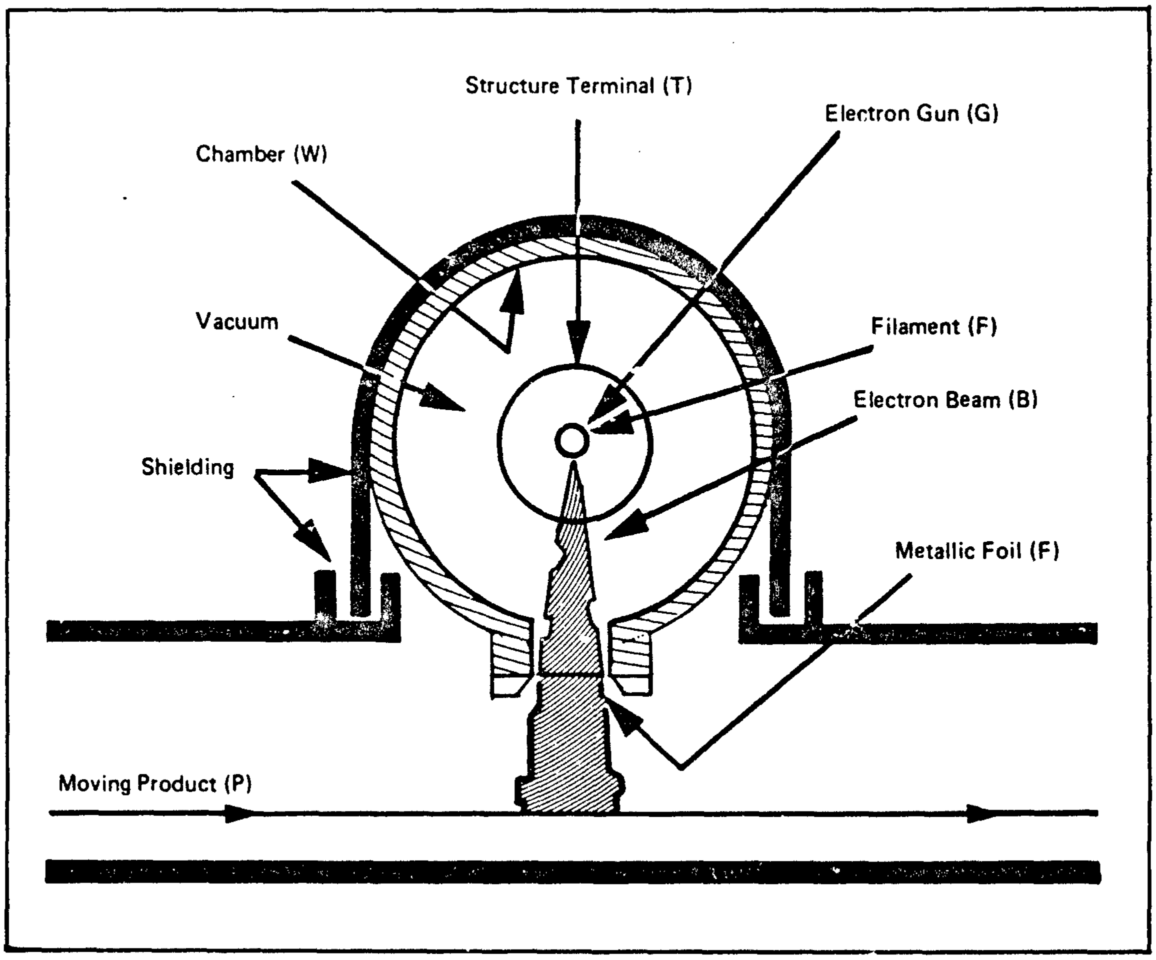


FIGURE IV-1 SCHEMATIC OF ELECTRON PROCESSOR

absorb the x-rays generated with the bombardment of matter by energetic electrons.

Electrons are accelerated in vacuum, and then emerge through the window and into air in the process zone. Since energetic electrons generate ozone and oxides of nitrogen in the air, these gases must be prevented from entering the work area. This is generally accomplished by the introduction of inert gas or pure nitrogen in the process zone, or by ventilation of the shielded process zone.

EB processors have been manufactured in many sizes from 54-in. sheet-fed systems for wood panel finishing to 72-in. web-fed systems for laminating and decorative coating to 6-in. tray-fed laboratory units for research and development.

3. Coating System

Almost any coating system, such as reverse roll or knife, can be used to apply the liquid EB coating to the substrate. However, the offset-gravure technique is generally used because its fixed metering ability is consistent with the economies and fluidity of many EB coatings.

4. Facilities

Metallized Products is unique in the industry because it has both metallizing and EB coating capability. Energy Sciences has a 72" EB coating facility in Woburn, MA, that is available on a toll basis. Any interested party could purchase an appropriate EB coating composition and contract for its application to aluminized Mylar. By the same token a laminator could do the same with his finished fabric to upgrade its abrasion resistance, although more problems would be involved in coating the contoured fabric laminate than in coating the smooth, flat, aluminized film.

E. ULTRAVIOLET COATINGS (UV)

These coatings are similar in composition to EB coatings with the exception that chemical sensitizers are added to make them specific to UV radiation. Such sensitizers absorb infrared energy as well as UV energy. Most of the sensitizers are not resistant to heat although some UV cured materials can be used briefly in a solder bath operating at about 500°F.

Therefore, after a few initial experiments with various types of general-purpose coatings, in which they burned up or lost adhesion to aluminum in the specified infrared test, we concentrated on high-temperature UV systems.

While one virgin UV-cured solder mask material narrowly avoided thermal decomposition in the specified IR test, it did not adhere adequately to the metallized surface (Table IV-3). Most of the UV coatings pulled off easily with Scotch tape or spalled off in the Wyzenbeck abrasion test.

F. PLASMA COATING

Plasma coating is a vacuum coating process in which organic molecules can be deposited on and polymerized on various substrates. Westinghouse Corporation has developed a process for coating small piece goods by this technique. Coated articles up to perhaps several feet square are the current limitation--Westinghouse claims to be able to engineer a continuous process. While this process is of no immediate interest, Dr. Cooke of Westinghouse, offered to coat several laminated samples with materials of interest to them.

The data in Table IV-4 show that a silicone material is of potential interest. That material, hexamethyl disiloxane, shows better abrasion resistance than the control fabric in the specified infrared test. It also adhered adequately to the aluminized surface as judged by a tape peel test.

We abandoned further work in this area because there is no commercial facility for producing the yardage required by our contract.

G. PARYLENE

Union Carbide Corporation has a vacuum process for evaporating paraxylylene and polymerizing it on the surface of many substrates. The process is currently limited to coating one-foot wide webs but Union Carbide claims to have sufficient engineering data to scale the process up to coat a web as wide as required by the firefighters construction.

The data in Table IV-5 indicate that Parylene coatings have considerable promise for protecting aluminized fabrics against mechanical and chemical abuse.

We did not pursue this concept any further because no source is capable of manufacturing the yardage required by the contract.

TABLE IV-3

ULTRAVIOLET CURED COATINGS

Resin	Adhesion to Aluminized Mylar	Specified Infrared Test
General Electric 1002	Poor	Burns
General Electric 1003	Poor	Burns
General Electric 1006	Poor	Burns
General Electric 1007	Poor	Flakes Off
Dynacure 5M110	Poor	Flakes Off
Dynacure 5M15	Poor	Flakes Off

TABLE IV-4

PLASMA COATINGS

Coating Material	Wyzenbeck Cycles to Failure
None (Control)	300
Hexamethyl disiloxane	600
Methyl methacrylate	< 600
Tetramethyl tin	< 600

TABLE IV-5

PARYLENE COATING

Thickness (mils)	Wyzenbeck Cyles to Failure	Resistance to Aqueous NaOH
None (Control)	300	< minutes
0.1	1800	> 24 hours
0.2	> 1800	> 24 hours

H. CONCLUSIONS

The data indicate that 100% solids coatings, such as EB, parylene or plasma, generally give better performance properties for the intended application than solvent applied coatings. Poor adhesion to the aluminized surface was noted in almost every solvent applied polymer. In several instances, thermal fusion of the dry material (after solvent evaporation) improved adhesion of the coating but in general the thermal fusion treatment tended to melt or degrade the supporting Mylar film.

Overall, EB coatings provide the best combination of adhesion, thermal resistance and abrasion resistance of the coating materials we examined. Furthermore, EB coating technology is available for producing the final product. Had Parylene technology been available for production, it probably would have been selected for exploitation because it appears to be considerably more resistant to chemical attack then EB coatings.

V. ALTERNATIVE FILMS FOR METALLIZATION

A. EXPERIMENTAL WORK

Early in the program we recognized that the Mylar film now being used for the application places a thermal limitation on the product. Mylar melts at about 250°C and softens almost 50°C lower. We believe that the Mylar film melts in the IR test when sufficient surface aluminum is removed by physical or chemical means. The molten Mylar then disrupts the back layer of aluminum to allow penetration of IR energy into the Kevlar fabric and beyond. In a typical IR test of abraded fabric a glowing spot, corresponding to the abraded area, can be observed to increase in brightness and size during the 30-second test--i.e., the melting process.

This observation led us to investigate films that have higher melting points than Mylar. A film that would not melt in the specified IR test would provide better performance than Mylar for the aluminized layer adjacent to the adhesive and fabric. Indeed, the film would act as a protective coating for the underlying aluminum. Of course, the overlying film would absorb heat in proportion to its thickness and spectral response but if it did not melt the underlying reflective aluminum would remain intact and protect the fabric and other layers to some extent. Under those conditions, the overlying film would protect the reflective layer for long periods against mechanical and chemical abuse and give a relatively constant thermal radiation protection although at some reduced level compared to the virgin unprotected metal found on the existing two-side aluminized Mylar.

Experiments were carried out by laminating the aluminized films, metal side in, to Kevlar cloth using a solid transfer film of silicone pressure-sensitive adhesive. The adhesive was selected because it will not char below about 450°F. While the adhesive is thermally stable and suitable for the experiments, it will not meet specifications for peel strength. However, the laminate is sufficient for evaluating thermal resistance of metallized films and for conducting abrasion tests.

The films were obtained from commercial sources and metallized by one of several industrial sources. Although we obtained the thinnest available commercial films some were as thick as three mils. Because most of the higher temperature resistant materials are produced by new extrusion technology, the films represent current state of the art. We anticipate that films of more appropriate thickness, i.e., < one mil, will be available in the future.

We stress the thickness aspect because the heavier films lead to an unacceptably stiff laminate. In addition, thinner films are consistent with lower heat absorption in a given chemical structure.

The data in Table V-1 indicate that any film having a melting point above 300°C is potentially useful for reflective fabrics. As expected, all of the high-temperature resistant films allow more heat buildup behind the construction than two-side aluminized Mylar. However, such new constructions guarantee almost constant radiant protection in contrast to the fast abrading existing product.

In these experiments we first tested virgin products in the specified visual IR test and also measured temperature behind the laminate for more quantitative data. Those virgin constructions that passed the IR test were abraded on the Wyzenbeck to failure in the specified IR test or, in the cases of polyether imide and polyimide films, until the adhesive bond failed. While the virgin control, i.e., the Gentex product, initially offers superior performance its longevity is much less than that of the polyimide film which has ten times as much abrasion resistance.

B. CONCLUSIONS

Polyimide films such as Kapton are available in half- and quarter-mil thicknesses which produce a laminate similar in stiffness to the existing product. While the polyether imide film apparently offers a second choice for a deliverable product, we were unable to obtain less than a two-mil thick film which resulted in an unacceptably stiff laminate.

On that basis, we selected half-mil polyimide films for metallization and lamination to deliver the required yardage. However, the other promising films should be reviewed when they become commercially available in lower gauges because they may have technical and economic advantages over Kapton.

TABLE V-1

PERFORMANCE OF SINGLE SIDE ALUMINIZED LAMINATION

Film Base Material	Apparent Melting or Decomposition Temperature (°C)	Specified Infra-Red Resistance (Virgin Condition)		Wyzenbeck Cycles to Failure
		Passes Test	Temperature Behind Fabric (°C)	
Polyethylene	130	Fails	> 500	--
Polypropylene	170	Fails	500	--
Nylon	210	Fails	500	--
Polyester	250	Fails	500	--
Polycarbonate	275	Fails	500	--
Polymethyl pentene	280	Fails	425	--
Polysulfone	300	Pass	300	1200
Polyether sulfone	325	Pass	325	1500
Polyether imide	325	Pass	325	> 1500
Teflon	> 350	Pass	230	< 300
Polyimide	> 350	Pass	350	> 3500
Control (Two-side Aluminized Mylar)	250	Pass	225	300

VI. SCALE-UP WORK ON BEST CANDIDATE FOR DELIVERABLE PRODUCTS

As discussed in the preceding section, we identified an EB coating for protecting two-side aluminized Mylar and a heat-stable film that overlays the reflective aluminum layer. Each requires a different production process and will be discussed separately. Preliminary trial runs under commercial conditions were carried out with the EB-coated product to assure ourselves that the concept could be scaled up from bench experiments. However, similar scale-up work was not feasible with the Kapton product because the problems of identifying an adhesive occupied our efforts until it was too late in the program to schedule preliminary on-machine lamination trials. The final deliverable products were laminated by the Gentex Corporation using the best available adhesive technology.

A. ELECTRON-BEAM COATINGS FOR PROTECTING TWO-SIDE ALUMINIZED MYLAR

Our initial laboratory work showed that several commercially available and proprietary electron beam coatings had promising properties of adhesion, IR resistance and abrasion resistance. We extended that work by making a trial run at the Energy Sciences facilities in Woburn, MA. Aluminized film corresponding to the film used in the existing laminated product was used for the coating trial and the final delivered product. The trial products were laminated by Gentex Corporation to produce about ten yards of 20-inch wide material.

The data in Table VI-1 show that all machine laminated trial products outperformed the commercial control in terms of abrasion and chemical resistance. We later determined on Gentex machine laminated material that MOBIL 204 coating at 3.9 pounds of coating per 3,000 square feet had the best adhesion to the aluminum after a water soak test.

B. KAPTON

Kapton is the commercial trade name of the polyimide film produced by E.I. du Pont de Nemours. At the time we decided to use this material for a deliverable product, no adhesive was identified. Two approaches were pursued to identify an adhesive prior to making the production run.

First, we solicited the help of Du Pont's Kapton Division and their adhesive division. Their final adhesive selection was:

TABLE VI-1

EXPERIMENTAL ELECTRON BEAM COATINGS[1]

Coating Material	Coating Weight (lb/200 sq ft)	Performance in IR Test: Abrasion Cycles to Failure (Accelerated Wyzenbeek)	Performance in IR Test: After Chemical Treatment[2]
Mobil 204	1.4	> 600	no failure
Mobil 204	3.9	> 1200	no failure
Metallized Products 108-48	1.4	> 600	no failure
Metallized Products 108-48	3.5	6-1200	no failure
None (Laboratory Control)	0.0	3-600	fails
None (Commercial Gentex)	0.0	< 600	fails

[1] Applied to the 2x aluminized 1/4-mil Mylar that corresponds to current product

[2] 30-second exposure to 0.1N NaOH

Polyester, Du Pont 56065	20.0 parts
Isocyanate, Du Pont RC803	1.0 part
Araldite 609, Ciba	0.8 part
Trichloroethylene	10-15 parts

Second, Gentex Corporation laminated several samples with a proprietary adhesive that was used for the deliverable product.

While both systems appear to have adequate dry and thermal bond strength, both products could be peeled apart at the Kapton metal interface after soaking in hot water. That is, the clear film parts from the construction and leaves metal adhering to the adhesive on the Kevlar fabric. Because this problem seemed to be generic to Kapton in spite of the adhesive used, we again requested assistance from Du Pont.

The unofficial consensus from several Du Pont sources was that the water absorption characteristics of Kapton are probably the cause of wet delamination. Kapton absorbs about 2% liquid water and it is presumed that the film physically swells by that amount. In contrast, other high- temperature films absorb much less moisture and it may be that the promising polyether imide resin, General Electric's Ultem, would have been a better candidate in terms of wet adhesion, if that film had been available in the desired thickness.

1. A Possible Adhesive Alternative

Arclad DEV 559X4220, Adhesives Research, Inc., Glen Rock, PA, was used throughout the research stage for the Kapton products. The adhesive is thermally stable in the specified IR test. However, this adhesive is probably not suitable for field use because it creeps slightly under repetitive mechanical stress allowing the film to crease. When the film creases on the laminate that area wears badly. In addition, the Arclad adhesive has a low peel strength which might lead to large-area delamination if the film were punctured.

2. Metallization

In preparation for producing the required yardage of the experimental Kapton construction, we faced a dilemma in deciding how to specify a trial lot of aluminized Kapton film because not all commercial aluminized Kapton samples would pass the IR test. We attributed the best performance of aluminized Kapton to either its aluminum level or a minimum of "windows" or microscopic skips in the metal.

To resolve that problem, we aluminized Kapton film to the levels shown in Table VI-2. Much to our surprise, only the lowest level of aluminization failed the specified IR test but all samples produced a

TABLE VI-2

ALUMINUM LEVEL VS. IR PERFORMANCE

Source	Aluminum Thickness (Angstroms)	Specified IR Test	Temperature behind Fabric (°F)
commercial	204	passes	340
commercial	763	fails	>600
commercial	2198	passes	320
ADL	80	fails	>600
"	221	passes	360
"	796	"	310
"	982	"	330
"	2007	"	300

temperature of about 300-360°F behind the fabric during the tests. Clearly, aluminum thickness was not a primary variable in failure.

We next viewed all the aluminized Kapton films at 100X magnification and found that all commercial and experimental samples contained numerous "windows" or skips in the metal. In a qualitative sense, we ranked the samples that failed the specified IR test as having more "windows" than those that passed.

In talking with commercial sources, we found that no one will guarantee freedom from "windows" and that each manufacturer claims to minimize the problem by proprietary means.

We, therefore, contracted with Metallized Products for Kapton film metallized to >200 angstroms and as free from "windows" as the state of the art would allow. The delivered product has an acceptable level of aluminum but it is less than desirable in terms of "windows." We learned in subsequent conversations, that "windows" are a variable problem and that they may occur randomly throughout a production run. The product could be aluminized a second time in an attempt to cover the primary "windows." However, we elected not to do so because a second layer and more aluminum might create adhesive problems.

VII. DELIVERED PRODUCT

A. SUMMARY

On the basis of the preceding work we selected two products for production runs of 150 yards each to be delivered in fulfillment of our contract. The two products were selected because both laboratory and pilot samples showed significantly better abrasion and chemical resistance then the standard product and because both retain their inherent reflectivity to infrared radiation over a wide range of abuse.

As stressed in this report, the experimental products represent a trade-off between reduced reflectivity and longevity of performance. The experimental laminates absorb more heat then the virgin existing product but maintain their own steady degree of reflectivity far better than the existing product under laboratory test conditions.

Even though the experimental products have some deficiencies in terms of specified laboratory tests, we believe they represent promising concepts that commercial vendors can improve upon. There is reason to believe that commercial vendors may be able to rectify the specified test deficiencies in the EB coating and indeed some of these deficiencies may prove not to be important in field tests of the uniforms constructed from the experimental laminations.

In particular, it is difficult to reconcile the superior abrasion resistance of the EB coating with its specified test deficiency that indicates poor coating adhesion. Considering that the EB coating has excellent adhesion throughout the abrasion test, we question the significance of the specified tape adhesion test.

B. PRODUCTION OF EXPERIMENTAL DELIVERED SAMPLES

1. Electron-Beam Coated Laminate

The electron-beam coated product was prepared by Metallized Products, Inc., Woburn, MA. They applied 3.9 lb/3000 sq ft of Mobil Electron Beam Coating #204 to the two-side aluminized Mylar that is normally used by Gentex Corporation for the existing product. The Mobil 204 coating was cured at the recommended dosage.

The EB-coated film was laminated to the Kevlar fabric by Gentex Corporation using the same adhesive as that employed on the control fabric. The Kevlar fabric, supplied by NCTRF, Natick, MA, is about 90 gms/m lighter than the original laminated fabric furnished to us as a control. This lower-weight fabric may have put the experimental

products at a disadvantage in comparison to the control in the heat transmission tests.

Unfortunately, the EB-laminated product is marred with a number of creases caused by mechanical difficulties on Gentex's combining operations. This is an operating problem, not an inherent characteristic of the coated film.

2. Kapton Product Laminate

A half-mil Kapton film was aluminized by Metallized Products with the request to produce as few "windows" as possible in the aluminized layer. This company was chosen because of its reputation and because its off-the-shelf samples showed a minimum of pinholes. Unfortunately, the roll furnished us had numerous pinholes which undoubtedly detracts from performance of the material.

The aluminized Kapton film was laminated by Gentex Corporation to the fabric furnished by NCTRF, Natick. The adhesive was a proprietary Gentex product because we saw no advantage in introducing the Du Pont adhesive into an operation not under our control.

The production roll was in excellent mechanical condition unlike the EB product.

C. STANDARD ACCEPTANCE TESTS

The experimental delivered products were tested in accordance with MIL-C-87076 against the heavier sample product delivered to us at the beginning of the program. The data, presented in Table VII-1, show that none of the products passes all the tests.

- The control product exceeds the maximum allowable weight.
- In the electron-beam coated product the metal adheres well enough to the Mylar film prior to the coating operation to pass the tape test. However, after the aluminized film is coated with EB 204 and cured with electron-beam energy, the tape test peels some coating and aluminum off the Mylar film. That phenomenon was noticed in the preliminary EB trial with three out of four coatings. We find that the production trial did not completely reproduce the preliminary trial because the production run of Mobil 204 coating peeled aluminum from the Mylar, whereas it had performed adequately in the preliminary trial.
- The experimental Kapton coated product delaminates upon wet flexing. We authorized the production run of Kapton anticipating that the product would be deficient in the wet-flex

TABLE VII-1

STANDARD ACCEPTANCE TESTS

Property	Control	Experimental EB Coated	Kapton
Weight (gms/m^2)	457 (fail)	363 (pass)	354 (pass)
Reflectivity after abrasion	pass	pass	pass
Adhesion of coating (tape peel)	pass	fail	pass
Stiffness (Taber units)	44	38	36
Wet flexibility	pass	pass	fail
Adhesion of coating after wet flexing	pass	fail	fail
Blocking	pass	pass	pass
Flame resistance	pass	coating cracks	pass
Flexibility at low temperature	pass	pass	pass
Flexibility after IR test	pass	pass	pass

test but that it would demonstrate a principle for further investigation. We suggest that if a polyether-imide film of appropriate thickness becomes available, the wet adhesion problem will disappear!

D. ADDITIONAL TEST METHODS

Although MIL-C-87076 describes a series of performance tests, we carried out other tests for research purposes. In particular, we investigated several ways of quantifying the subjective IR test in that document, because "pass on fail" observations are of limited value for research purposes.

1. Accelerated Wyzenbeck Testing

The specified Wyzenbeck test uses a low vertical loading to abrade the fabric. Because of the fabric contours only the uppermost areas of the aluminum are worn off the twill fabric, but the aluminum in that location is worn off long before the specified cycles are completed. Visual observation clearly shows spots of transmitted light corresponding to the tips of the twill fabric. We conclude that most of the test is devoted to abrading Mylar film because the abradant never really contacts the valley(s) of the aluminized surface. In fact, the control construction barely fails the IR test no worse at 300 than at 3000 cycles in the specified Wyzenbeek test.

We therefore increased the loading on the Wyzenbeck tester from a nominal 3 pounds to 15 pounds to insure more uniform contact. In addition, we placed a 1-inch by 6-mil thick tape on the Wyzenbeek drum to further intensify wear on a specific area. This not only speeded up the testing and enabled us to discriminate between improvements but also simulated actual conditions more closely e.g., the uniform wearer's elbow, knee or posterior rubbing hard on some surface in the field.

These modified test conditions described are identified as accelerated Wyzenbeek Testing ("W_A").

In Table VII-2, we show a correlation between the specified Wyzenbeck Test and our accelerated "W_A" test for three constructions.

TABLE VII-2

ACCELERATED WYZENBECK TESTS

Construction	Cycles to IR Failure: Specified Wyzenbeck Test	Cycles to IR Failure: Accelerated Wyzenbeck Test (W_A)
Control	> 300	3-600
EB Coated	> 600	> 1800
Kapton	> 600	> 3000

2. Infrared Resistance After Wet Flexing

Although the control and the experimental EB product pass visual inspection for absence of delamination in the flex test, neither product will pass the specified IR test after the wet flexing test. Evidently the wet flexing test disrupts the continuity of the aluminum films common to both products.

3. Infrared Resistance after Crinkling and Crushing

None of the products we have examined will pass the specified IR test after they are crushed numerous times by hand. This appears to be due to cracking of the aluminum film which can be readily observed with transmitted light at low magnification.

4. Chemical Resistance

The thin layer of exposed aluminum on the control laminate is vulnerable to chemical attack by firefighting chemicals, such as Purple K, salt spray and other chemicals that may be found in the environment of use. Almost any coating over the aluminum will inhibit or delay the attack of chemicals on aluminum.

We tested the chemicals shown in Table VII-3 for their rate of attack on the outermost layer of the reflective surface of the films used to construct the various laminates. Drops of various solutions were placed on the test film for different times in a 90°F, 90% RH test chamber. After cleaning off the residual chemicals at the specified time with a lens tissue, we evaluated chemical attack by viewing the aluminized film with transmitted light. Any difference in the transmission of light in the treated area clearly shows attack on the reflective layer.

The data show that Kapton protects its underlying layer of reflective aluminum from chemical attack better than the EB coating. We believe most of the problem with EB product is related to pinholes or microscopic voids in the coating. Clearly both experimental products protect better against chemical attack then the naked aluminum layer on the control fabric.

5. Infrared Reflectance Spectra

The measurement of the infrared reflectivity of opaque aluminized materials is complicated when the metal is overcoated with an organic coating and even more so when the topography of an underlying fabric is introduced. Total reflectance, as measured with an integrating sphere, is the only true way to avoid misleading specular components of the radiant energy. Unfortunately, total reflectance can be measured only in the visual and near infrared. No available

TABLE VII-3

CHEMICAL RESISTANCE OF ALUMINIZED FILMS

Substance	Time (hours)	Visible Change in Reflective Surface		
		Control	Electron Beam	Kapton
H_2O	24	No	No	No
	4	No	No	No
	0.1	No	No	No
NaCl	24	Slight	No	No
	4	Slight	No	No
	0.1	No	No	No
K_2CO_3	24	Yes	No	No
	4	Yes	No	No
	0.1	No	No	No
$NaHCO_3$	24	No	No	No
	4	No	No	No
	0.1	No	No	No
NaOH	24	Yes	No	No
	4	Yes	No	No
	0.1	Yes	No	No
Acetic	24	No	No	No
	4	No	No	No
	0.1	No	No	No
$KHSO_4$	24	No	Yes	No
	4	No	No	No
	0.1	No	No	No
HCl	24	Yes	Yes	No
	4	Yes	Slightly	No
	0.1	No	No	No
Motor Oil	24	No	No	No
	4	No	No	No
	0.1	No	No	No
Grease	24	No	No	No
	4	No	No	No
	0.1	No	No	No
Paint Thinner	24	No	No	No
	4	No	No	No
	0.1	No	No	No
Tide Detergent	24	No	No	No
	4	No	No	No
	0.1	No	No	No

TABLE VII-3 (continued)

Substance	Time (hours)	Visible Change in Reflective Surface		
		Control	Electron Beam	Kapton
Ansul AFC-5A	24	No	No	No
	4	No	No	No
	0.1	No	No	No
Ansul AFC-5	24	No	No	No
	4	No	No	No
	0.1	No	No	No

instruments provide similar measures in the region lying above 2.5 microns in the infrared region of the spectrum.

The data in Figures VII-1 and VII-2, show the reflectivity of the metallized control film, EB-coated film and Kapton over the range of 1-22 microns when the measurements are made on non-integrating IR instrumentation. The curves were subsequently integrated mathematically for a 2730°F fire to suggest the following total reflectivity:

	$\bar{R}$ (%)
Control film (no coating)	0.90
Electron-beam coated	0.75
Kapton (film over metal)	0.88

While those data are valid for that particular set of measurements, we do not believe they represent real life because the EB-coated product absorbs less heat than the Kapton product. Repeated thermocouple and calorimeter measurements show that Kapton constructions transmit more heat than the electron beam coating in the IR test -- that is, Kapton is not as reflective as the data indicate.

Next, we measured total reflectivity of the above films laminated to Kevlar cloth using an integrating sphere to compensate for the topography of the laminate. The data shown in Figure VII-3 qualitatively suggest that the Kapton construction has somewhat less favorable reflectivity than the control or the electron-beam coated construction in the visible and near infra-red regions.

Again, we question the real life validity of those data because they do not include any radiant energy above 2.7 microns and because calorimeter and thermocouple measurements show Kapton to absorb considerably more heat than the other two test materials.

6. Thermocouple Measurement

For a while, we used thermocouples to measure the temperature on the fabric side of various laminated constructions in order to judge relative efficiency in the IR test. Such measurements are valid and reproducible (±10°F) when the surface is uniformly reflective.

However, the surface of abraded laminates is not uniform because discontinuities are produced during the abrasion test and these cause wide variations in recorded temperature depending upon thermocouple placement. Because the abrasion defects are not readily visible, it

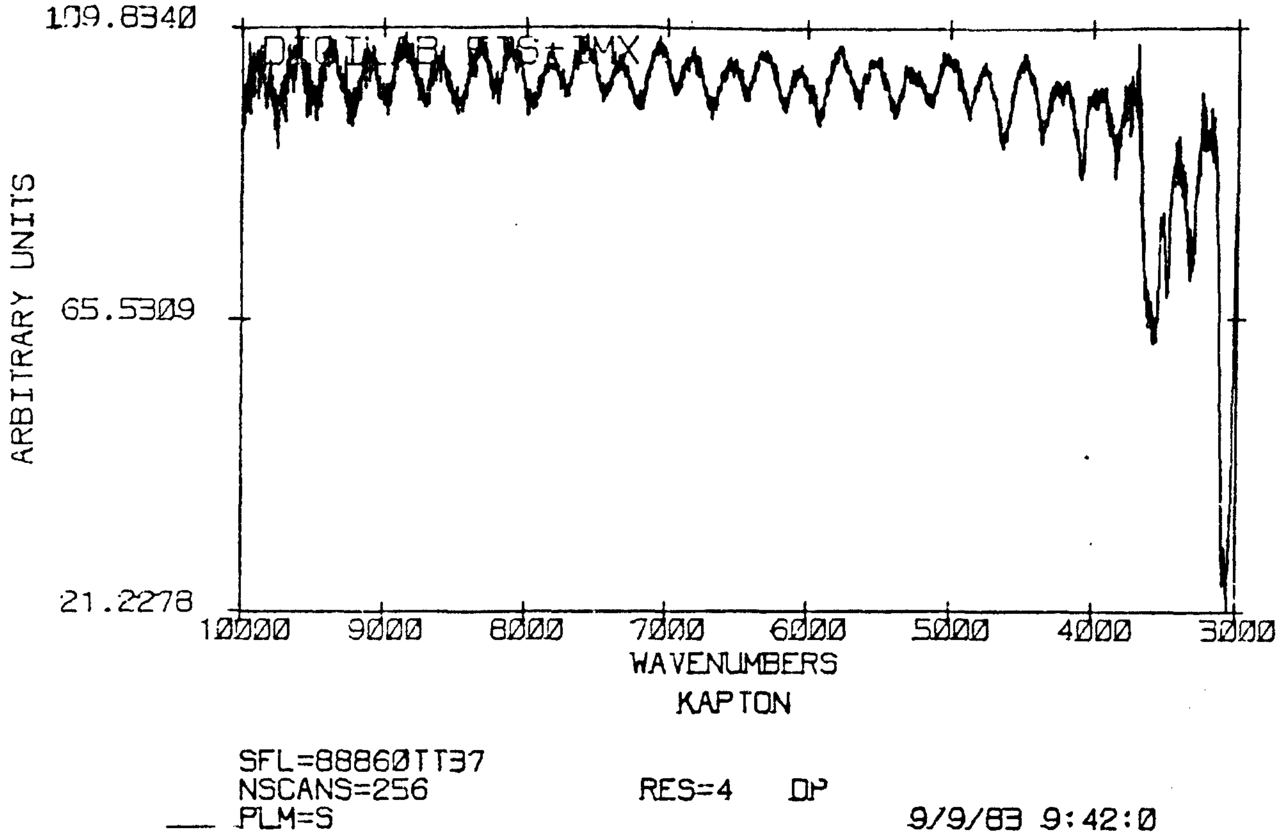
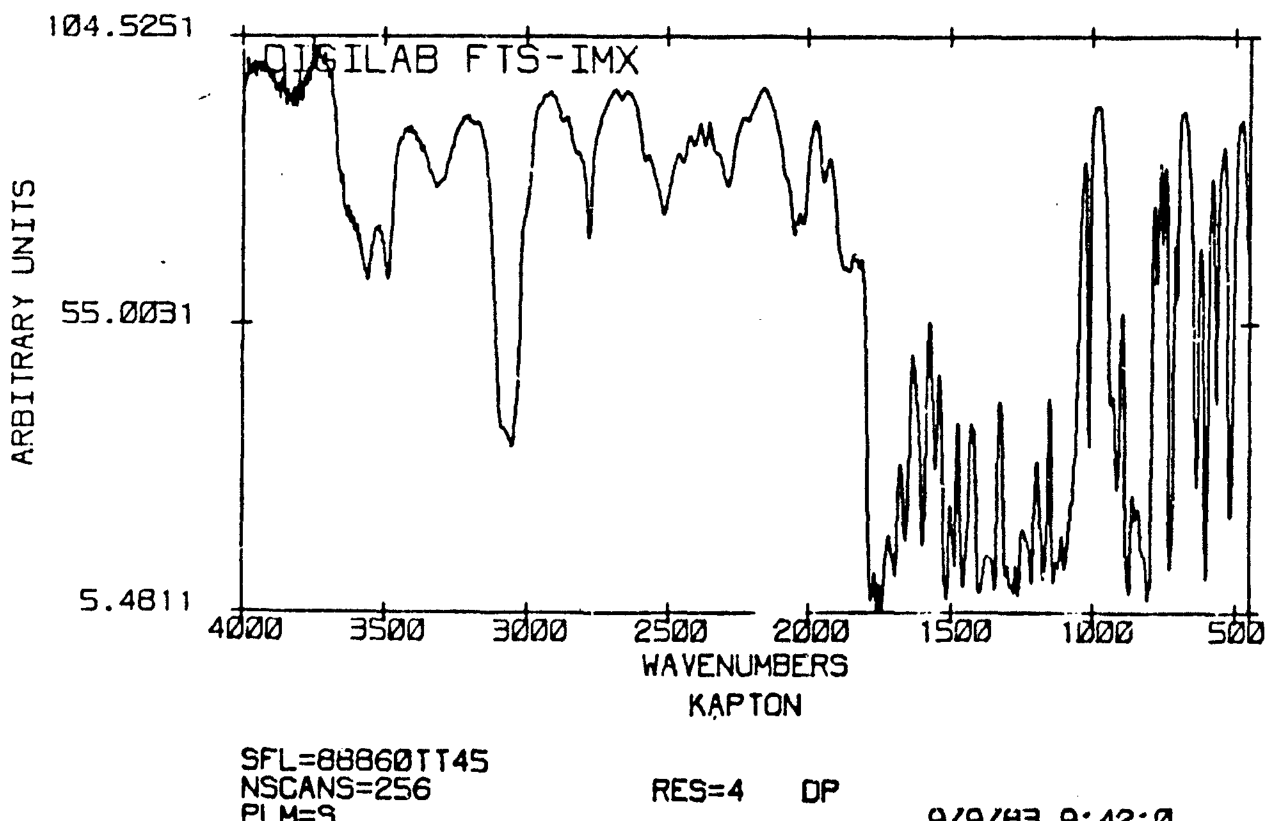


FIG. VII-1

TOTAL REFLECTIVITY - KAPTON



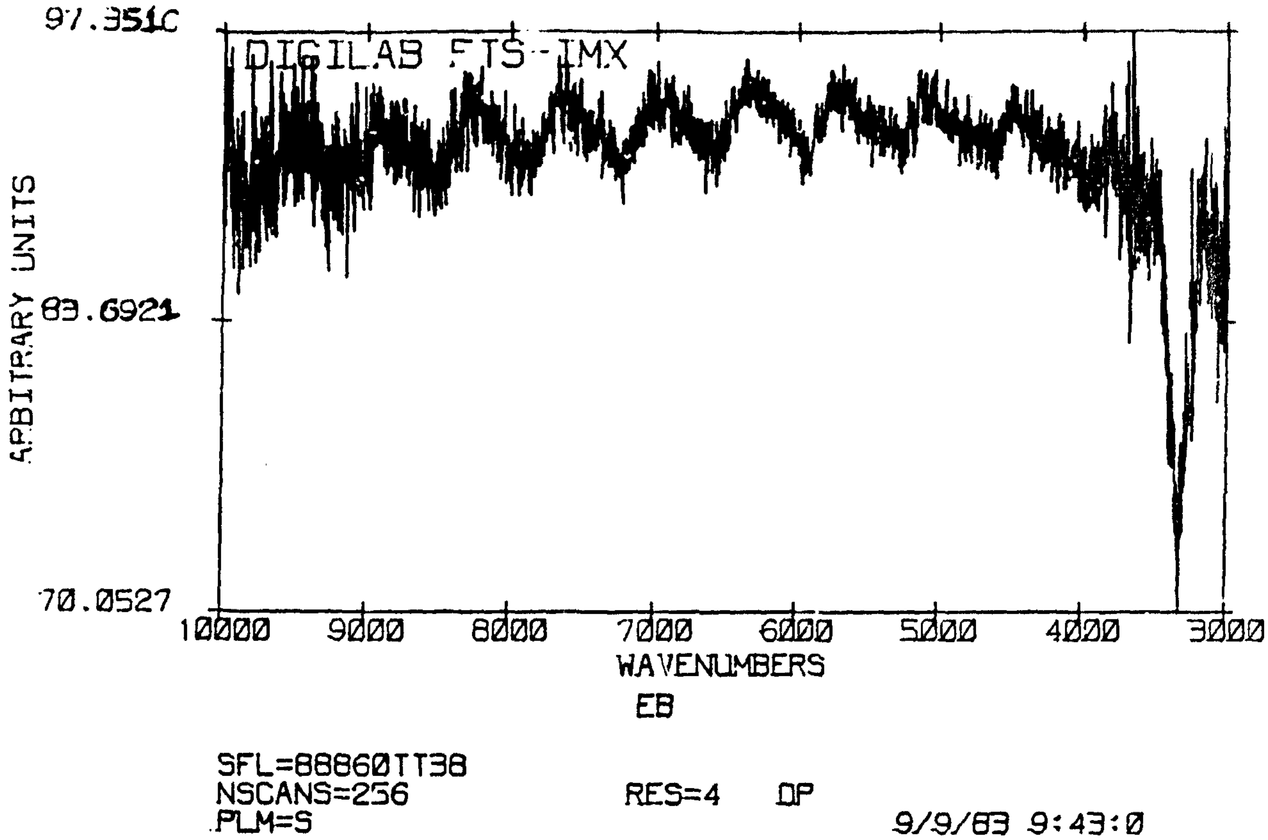


FIG. VII-2

TOTAL REFLECTIVITY - EB-COATED FILM

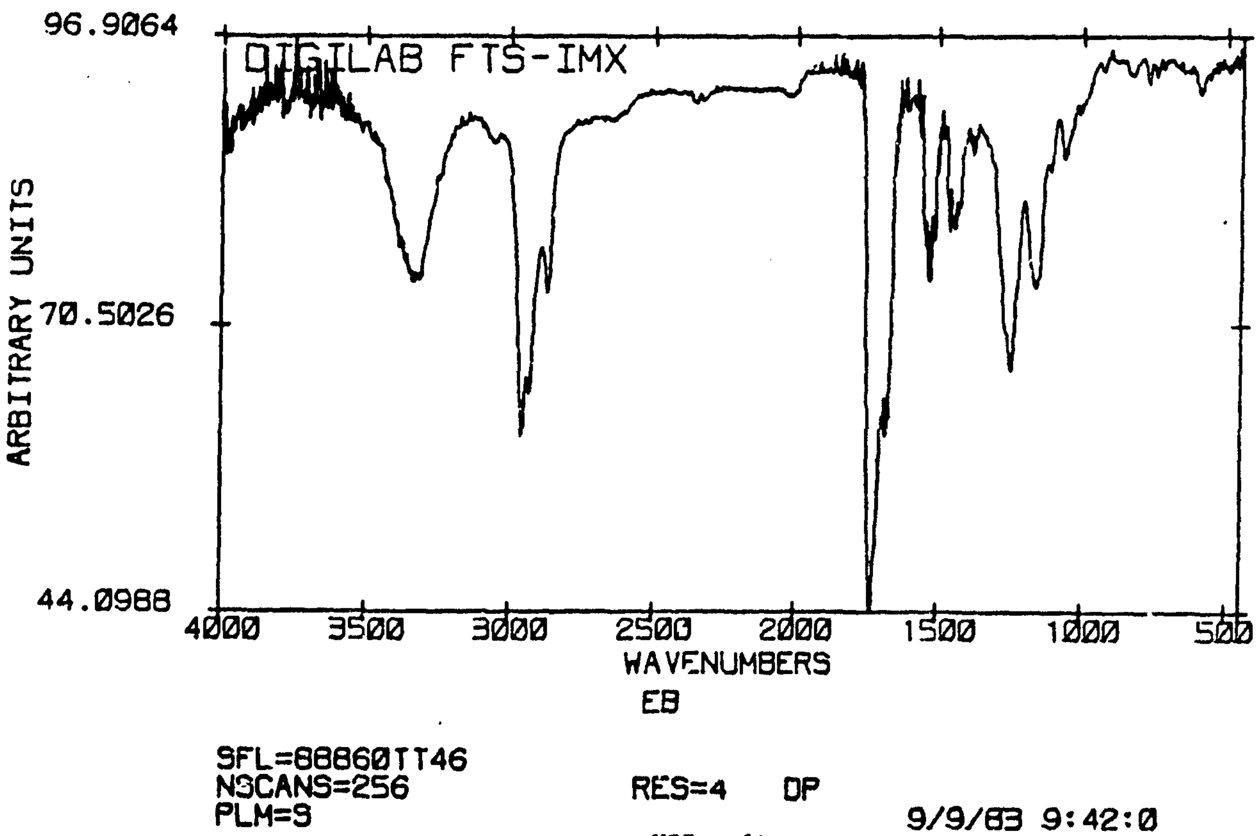
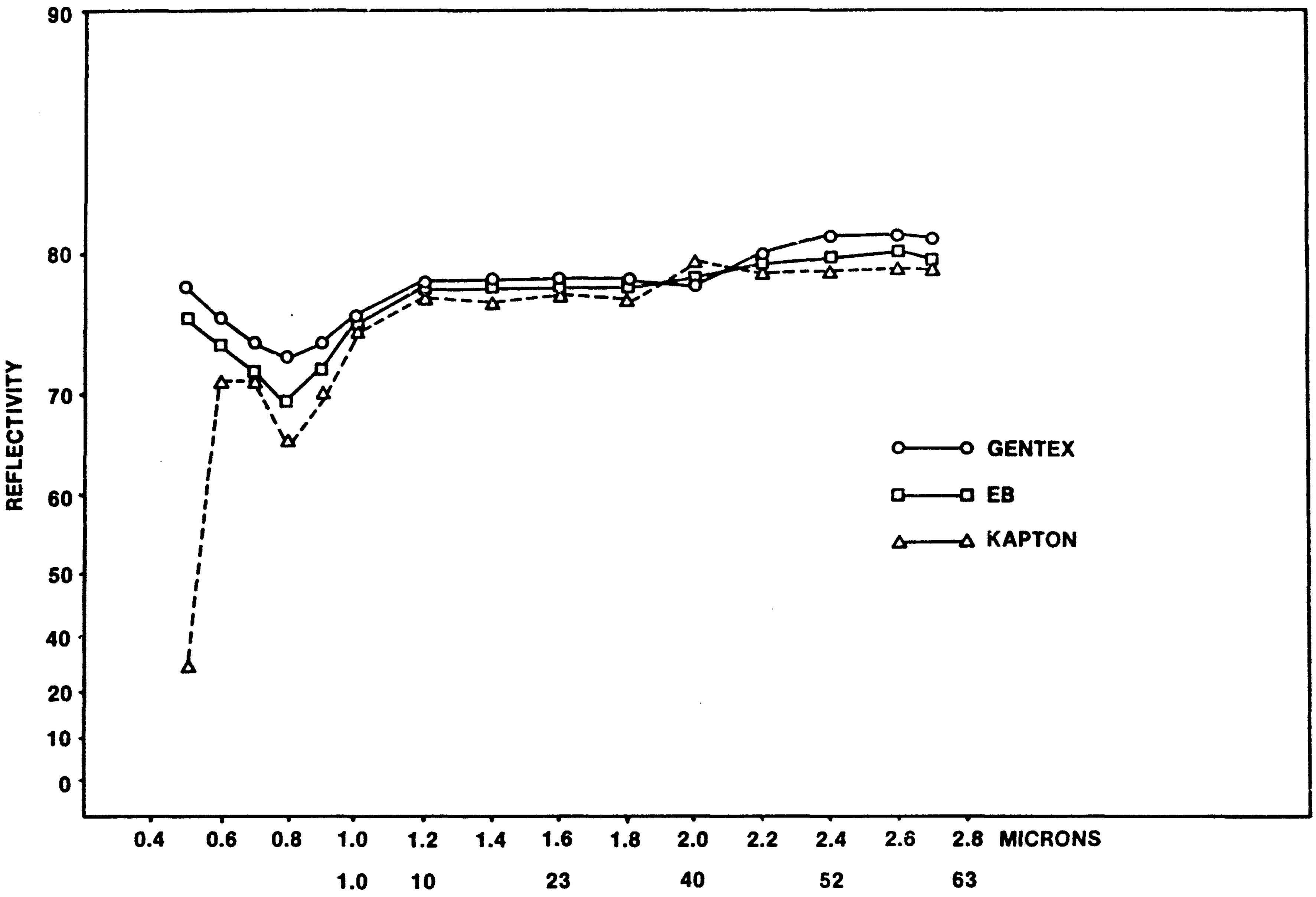


FIG. VII-3

TOTAL DIFFUSE REFLECTION OF LAMINATES



is almost impossible to place a thermocouple directly beneath the defect.

The results of two sets of experiments are shown in Tables VII-4 and VII-5. Overall we feel the temperatures behind virgin construction are representative of our measurement. The temperatures behind an abraded fabric are subject to wide variation caused by local wear and thermocouple placement.

7. Calorimeter Measurements

a. Laminates

Because of the thermocouple placement problems described earlier and because the problem is more concerned with heat absorption than with a spot temperature, we searched for a simple, easy-to-use, calorimeter-like device. In essence, any metal block of known heat capacity (sized to correspond to the abrasion test area) together with a temperature indicator would suffice to measure absorbed heat.

One such device is available from Optical Engineering Inc., Santa Rosa, CA. It is a black metal body equipped with a dial thermometer calibrated in watts. The device is intended to absorb and measure laser power but it will measure heat when placed behind a reflective laminate exposed to radiant energy. When exposed directly to the radiant IR source it will also measure power received by the reflective laminate. Therefore, the efficiency of a reflective laminate can be easily calculated.

The data in Table VII-6 show the relative efficiency of various laminates in both virgin and abraded condition when exposed to the specified IR source. Clearly, the virgin laminate reflects

TABLE VII-4

TEMPERATURES[1] ENCOUNTERED IN IR TEST OF COMPLETE CONSTRUCTION (°F)

Reflective Layer	Behind Outer Reflective Layer		Behind Inner Quilted Layer	
	Virgin	Abraded	Virgin	Abraded
Control	267	480[2]	144	159
EB Coated	389	407	173	178
Kapton	400	400	205	210

[1] Measured with iron/constantin thermocouple.

[2] Middle raincoat layer melted and scorched.

TABLE VII-5

TEMPERATURES[1] IN FULL CONSTRUCTION (°F)

Laminate	Virgin Behind Laminate	Virgin Behind Quilt	Abraded Behind Laminate	Abraded Behind Quilt
Control Gentex	267	144	480 (laminated char, raincoat melt)	159
EB Coated	389	173	407 (no visible damage)	178
Kapton	400	180	410 (no visible damage)	185

[1]Measured with iron/constantin thermocouple

TABLE VII-6

REFLECTIVITY[1] DIRECTLY BEHIND ALUMINIZED LAMINATES
(%, radiated power)

Laminate	Unabraded	Abraded[2] 300 cycles	Abraded[2] 900 cycles
Control	87	69	53 (scorcer)
Electron-beam Coated	70	70	70
Kapton	64	64	64

[1] IR source = 118 watts @ test plane

[2] Accelerated Wyzenbeck Test

more radiant heat than the experimental constructions. Just as clearly, the data for the virgin laminates are in agreement with the theory of reduced reflectivity caused by putting an organic coating over the aluminum metal.

The electron-beam coated product maintains an almost constant response to radiant energy unit until the coating is destroyed and the aluminum is exposed. In contrast, the existing product progressively loses its reflectivity at a much faster rate. For example, at the point of mechanical wear of the existing commercial material enough radiant energy penetrates to damage or destroy the second layer of fabric in the uniform, the electron-beam coated product still maintains almost its original reflectance efficiency. We estimate that the electron-beam coated product has almost ten times the abrasion resistance as the existing product. Similarly, the electron-beam coated product resists attack by chemicals and solvents common to the Naval firefighter's environments much longer than the existing fabric.

Even more clearly, the experimental constructions guard against mechanical abrasion. The numbers, however, do not tell the whole story. As illustrated in Figure S-1, show the control absorbed enough heat to char the Kevlar fabric. In contrast, both experimental construction are almost unchanged even though they received the same amount of abrasion and radiant heat.

Therefore, the two experimental products trade off some added heat absorption for extended longevity of mechanical performance. In essence, they prolong the useful life of the fabric and delay the onset of a catastrophe that would melt or char the underlying components of a firefighters uniform. All in all, the experimental laminates promise better human protection.

b. Full Uniform Construction

We used the sample calorimeter technique described in Section C, above, to measure the heat present at the layer adjacent to the firefighter's skin during exposure to the specified IR source. The "calorimeter" was placed behind the innermost fabric and insulated from convective cooling with a second layer of quilted fabric.

The data in Table VII-7 show that all virgin laminates protect the firefighter against much of the radiant energy. The data show that the EB-coated and Kapton products preserve that protection much longer than the control when subjected to mechanical abrasion.

TABLE VII-7

HEAT[1,2] BEHIND LAMINATE IN IR TESTS (WATTS)

Abrasion (Wa Cycles)	Control	EB	K
Virgin	18	32	42
300	34	34	44
600	51 (scorch)	39	44
900	53 (scorch)	42	46
1800	68 (char)	49	48
3000	75 (char)	49 (no visible damage)	49 (no visible damage)

[1]Measured with an calorimeter

[2]Radiation at surface of laminate - 118 watts

8. Other Abrasion Tests

We examined a number of alternative abrasion tests because the Wyzenbeck testing machine is almost obsolete and results only in comparative data specific to abrasion against cotton twill. In fact, the metal on the control laminate can be easily removed from the control fabric with anything from a soft toothbrush to an eraser to a polishing cloth. For example, early in the program we waxed the control aluminized fabric with floor polish in the hope that it might provide lubricity and prevent abrasion. Much to our dismay, most of the surface aluminum ended up on the soft polishing cloth.

While any standard abrasion method will show qualitative differences between, or among, experimental reflective coatings, the real problem is how to measure the amount of aluminum removed and how to correlate the abrasion test with IR test performance. For example, a Taber Abrasion Test uses a 4½" disk sample which is the wrong size for carrying out the specified IR test. It would be easy to measure aluminum lost in the Taber test by light transmission through the abraded area but, again, there is no correlation with the specified IR test.

Such a light transmission measurement for loss of aluminum could probably be correlated with IR reflectivity with sufficient work but that was beyond the scope of this assignment.

We came to similar conclusions after some experimentation with the Stoll Abrasion Test. Because each abrasion test uses a specific geometry, condition, etc. that is not easily compatible with the specified IR use test, we abandoned this effort.

Several experiments with a "falling sand" test indicated that aluminum is worn off the control film at a very rapid rate. Similar experiments with aluminized EB and Kapton films show complete protection of the metal far beyond the failure point of the control.

9. "Cleanability"

The "cleanability" of the experimental laminations was judged by soiling the reflective surfaces with backyard mud and a mixture of grease and backyard dirt. Both substances were easily removed by washing with a soft cloth and a mild dishwashing solution, e.g., "Joy".

No evidence of dulling of the surface or removal of reflective aluminum metal was noted with either the control or the experimental samples.

On the other hand, a scrub brush easily scratched the control surface but left no visible evidence of scrubbing on the experimental samples.

Therefore, we believe the experimental samples have better cleanability for washable soils than the existing product. However, we would not recommend any of the products with strong solvents, such as a lacquer thinner or paint remover, to clean up a garment that was accidentally soiled with paint, lacquer or any other film-forming material.

VIII. LITERATURE SEARCH

Our literature search revealed no information concerning the performance of coatings on highly reflective surfaces in the specified IR test. However, a considerable body of literature is concerned with the theory and developmental state of high temperature resistant flexible polymers that exceed the thermal resistance of Mylar either as a film, as an adhesive or as a coating. While some of these materials have been commercialized, many others are still in research or development stage.

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